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DRAWINGS ATTACHED

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(54) SELF-HEATING COMPOSITION

(71) We, THE GILLETTE COMPANY, a corporation organized and existing under the laws of the State of Delaware, United States of America, of Prudential Tower Building, Boston, Massachusetts, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed to be particularly described in and by the following statement:—

The present invention is concerned with self-heating compositions and more specifically with self-heating cosmetic compositions, such as aqueous shaving creams.

Self-heating compositions, such as shaving creams, are presently available in aerosol containers. Generally such compositions involve the use of a combination of heat-generating components which effect an exothermic reaction when in contact with one another. The aerosol containers which are used for such compositions generally comprise at least two separate compartments wherein the heat-generating components can be stored separately, along with the other ingredients, until time of use. Upon use, the heat-generating components are dispensed from the separate compartments through concurrently operated valves into a common discharge passageway where they come together to effect the exothermic reaction and thereby elevate the temperature of the composition. Although such aerosol-dispensed, self-heating compositions have met with considerable success, they require multi-compartment containers and close tolerance valving systems in order to store the heat-generating components separately and to dispense said components in the proper stoichiometric amounts under a myriad of operating modes and conditions. We have now developed self-heating compositions which eliminate the need for such multi-compartment containers and close tolerance valving systems.

According to the present invention, we provide a self-heating pharmaceutical or cosmetic composition, comprising a vehicle and a first and a second heat-generating component maintained separately from one another but which when in contact with one another effect an exothermic reaction; at least one of said components being encapsulated into a plurality of frangible capsules which are dispersed in said vehicle, the encapsulating material for said capsules being both inert and impermeable with respect to the vehicle and the heat-generating components.

In the self-heating compositions of the present invention the need for multi-compartment containers and close-tolerance valving systems is eliminated by encapsulating at least one of the heat generating components in frangible capsules and dispersing the capsules in the vehicle. The second heat-generating component may be added directly to the vehicle or it may also be encapsulated and dispersed in the vehicle. During use, the capsules are ruptured by mechanical force, e.g. by rubbing the composition in one's hands to release the heat-generating components and bring them together to effect the exothermic reaction. Generally, the present invention is broadly applicable to a wide range of hydrophilic and hydrophobic compositions. It is particularly useful in aqueous-based pharmaceutical and cosmetic compositions, such as shaving creams, wherein heat is desired to increase the effectiveness of the composition.

The present invention also comprises a single-use packet containing a self-heating composition, said packet comprising an enclosed chamber in which a composition according to the invention is dispersed, said chamber being bound by at least one resilient wall and having a dispensing opening which is closed off by a removable closure.

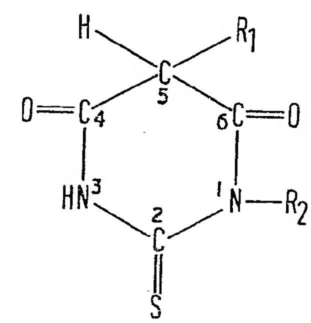
Processes for encapsulating the heat-generating components are known. As examples of such processes, mention may be made of chemical methods such as disclosed in U.S. Patents Nos. 2,800,457, 2,800,458 and 3,155,590, mechanical methods such as disclosed in U.S. Patents Nos. 3,015,128 and

3,423,489 and electrostatic processes such as disclosed in U.S. Patent No. 3,159,874. Generally, such processes comprise forming a continuous wall of the encapsulating material around the material to be encapsulated and thereafter solidifying the encapsulating material. A wide variety of substances have been found useful as encapsulating materials. Such materials, in addition to being capable of being deposited around the material to be encapsulated, also have to be inert and impermeable with respect to both the compositions in which they are to be dispersed as well as the encapsulated material. Generally, if the encapsulated material and the vehicle in which it is to be dispersed are hydrophilic, the encapsulating material will be of a hydrophobic nature and if the encapsulated material and the vehicle in which it is to be dispersed are hydrophobic, the encapsulating material will be of a hydrophilic nature. As examples of typical encapsulating materials, mention may be made of gelatin, ethyl cellulose, polymethylmethacrylate, wax, gum arabic, starch, paraffin, polyethylene, polyvinyl alcohol, and polystyrene.

Generally, encapsulation processes such as set forth above are capable of producing capsules having diameters varying from about a few microns up to about 4000 microns and even larger. In the compositions of the present invention, the size of the capsules may be chosen to fit the particular need. In embodiments wherein the capsules are intended to be ruptured by hand, the rupturing can be facilitated by using the larger diameter capsules, e.g. 2000 microns and above.

The heat-generating components for use in the compositions of the present invention may be broadly selected from the various combinations of materials which effect an exothermic reaction when in contact with one another. As can be appreciated, when the composition is to be topically applied, the heat-generating components and the by-products of the exothermic reaction should be non-toxic and non-irritating. As examples of types of heat-generating combinations, mention may be made of the combination of an acid and base and the combination of a solvent and a solute having an appreciable heat of solution or dilution in the solvent, e.g. the combination of water and ethylene glycol and the combination of water and salts such as aluminum sulfate, calcium chloride, copper sulfate, ferric chloride, magnesium chloride, and magnesium sulfate. The latter combinations may be conveniently used in aqueous-based composition by merely encapsulating the salt in a water-insoluble water-impermeable encapsulating material and dispersing the capsules in the composition.

In preferred embodiments of the present invention, the combination of an oxidizing reagent and a reducing reagent are used as the heat-generating components. Such oxidizing and reducing reagents may be selected broadly from the various compounds of this nature available. As examples, of oxidizing agents, mention may be made of chlorates, perchlorates, permanganates, persulfates, peroxides, nitrates, metal oxides, such as copper oxide, lead oxide, and iron oxide, and perborates. The preferred oxidizing agents include hydrogen peroxide, urea peroxide, sodium peroxide, sodium perborate, sodium persulfate, ammonium persulfate, potassium persulfate, and mixtures of any of two or more of the foregoing. As examples of reducing reagents mention may be made of metals such as magnesium, zinc, aluminum and iron; sulfites, thio-sulfates, thioureas, imidazolinethiones, thiotriazoles, thiopyridines, thiopyrimidines, thiols, thio-acids, sulfoxides, xanthates, ortho- and para-polyhydroxy benzenes, aldehydes, and glycols. A preferred class of reductants for use in compositions which are to be topically applied are the thiobarbituric acid derivatives of the formula:



in which R_1 may be hydrogen or an alkyl, hydroxyalkyl, alkoxy, or alkanoyl group, each of which may have up to 5 carbon atoms, and R_2 may be any of the foregoing except hydrogen and may in addition be phenyl, which are described in British Specification No. 1,088,301. Examples of such compounds are 1 - phenyl - 2 - thio - barbituric acid, 1 - phenyl - 5 - ethyl - 2 - thiobarbituric acid, 1 - methyl - 2 - thiobarbituric acid, 1 - methyl - 5 - methyl - 2 - thio - barbituric acid, 1 - methyl - 5 - ethyl - 2 - thiobarbituric acid, 1 - ethyl - 5 - ethyl - 2 - thiobarbituric acid, and 1 - phenyl - 5 - methyl - 2 - thiobarbituric acid.

Generally, the amounts of heat-generating components employed in the compositions will vary depending upon factors such as the heat of reaction of the components; the specific heat of the composition and the ultimate temperature desired. Usually, the heat-generating components will be used in

amounts which will provide a sensible temperature rise to the composition (e.g. a rise of 25°F from room temperature in a minute). As can be appreciated, the amounts which will produce such a rise will vary from system to system but can be readily calculated or empirically determined. In aqueous systems, such as shaving creams, when employing the heat-generating components disclosed in U.S. Patent No. 3,342,418 as little as 0.8% by weight of oxidant based on the total weight of the aqueous composition will suffice to produce a sensible temperature rise when a stoichiometrically equivalent amount of reductant is used. In preferred embodiments of such aqueous compositions, at least 1% by weight of the oxidant, with a stoichiometrically equivalent amount of the reductant, is employed.

When desired, catalysts or amounts of one of the heat-generating components in excess of the stoichiometric amount may be used to promote or accelerate the heat-generating reaction. When employing catalysts such materials may be separately encapsulated or they may be added to the phase of the composition in which they are inactive. As can be appreciated, the type of catalyst will depend upon the nature of the heat-generating components. As examples of catalysts which can be employed in oxidant-reductant systems such as disclosed in U.S. Patent No. 3,341,418 mention may be made of the alkali metal and ammonium salts of molybdates and tungstates.

In especially useful embodiments of the present invention, the compositions are self-foaming as well as self-heating. This is accomplished by incorporating into the foamable composition a foam-generating material which is insoluble or only slightly soluble in the vehicle of the composition e.g. less than 2 gms. per 100 cc and which is a liquid at room temperature, e.g. 25°C but boils at the elevated temperature which will result from the reaction of the heat-generating components. In preferred embodiments of the present invention, the foam-generating materials will have boiling points between about 95°F to about 160°F. As examples of foam-generating materials mention may be made of pentane, hexane, dichlorotetrafluoroethane and trichlorotrifluoroethane. When the compositions are to be marketed in single use, packets, or in containers which will be open to the atmosphere, only for short periods of time during the dispensing of the composition, the foam-generating materials may be added directly to the compositions. In uses wherein the compositions may be exposed to the atmosphere for extended periods, the foam-generating materials may be encapsulated. At time of use, the capsules containing the foam-generating materials are ruptured along with those containing the heat-generating components to provide a self-heating, self-foaming composition.

In preferred embodiments of the present invention, the heat-generating components are encapsulated in thermoplastic materials which have melting points at or below the temperature which will be generated in the compositions. Such embodiments make it possible to provide compositions which are able to generate heat over a prolonged period and thus may be called self-sustaining. Generally, in such self-sustaining compositions the heat-generating components are present in amounts in excess of that which is necessary to bring the composition to the desired or specified temperature. In using such self-sustaining compositions, at least that portion of the capsules is ruptured which will bring the composition or a portion of it to the temperature at which the walls of the unruptured capsules will melt. As the composition or portions of it reach such temperatures, the walls of unruptured capsules in the composition or adjacent to the heated portion of it will gradually melt over a prolonged period and continuously release additional quantities of the heat-generating components; thus sustaining the evolution of heat. In compositions which are self-foaming, as well as self-heating, the foaming action may be similarly sustained by also incorporating the foam-generating materials into similar capsules.

In still another preferred embodiment of this invention, the compositions are compounded so that they may be elevated to the desired temperature over a prolonged period. This is accomplished by incorporating the heat-generating components in a plurality of thermoplastic capsules which have sequentially higher melting points e.g. a first portion of the heat-generating components are encapsulated in an encapsulating material which melts at e.g. 120°F, a second portion is encapsulated in a material which melts at 140°F, and so on. Generally, the melting points of the encapsulating material will lie in a range of temperatures which are above the temperature at which the composition will normally be stored and at or below the desired temperature to which the composition is to be heated. When it is desired that the compositions also be self-sustaining, this is readily accomplished by using an excess of the heat-generating components and incorporating the excess components in the highest melting encapsulating material.

In certain end uses of the compositions of the present invention, e.g. cosmetics such as shaving creams, the fragments of the ruptured capsules give the compositions a gritty feel and are undesirable. Such undesirable characteristics may be substantially reduced by encapsulating the reagents in thermoplastic materials, such as mentioned above, which have melting points at or below the tempera-

ture which will be generated in the compositions by the reaction of the heat-generating components. In the melted state such encapsulating materials are usually readily dispersible in the compositions and thereby provide a convenient mode of eliminating the undesirable feel of the solid shell fragments. In some compositions, such as shaving creams, there will be surface active agents present which will facilitate such dispersion. In compositions where such surface active agents are not present, it is preferable, if the formulation will tolerate it, to add such surface active agents to aid in the dispersion.

Generally, the thermoplastic encapsulating materials for use in this invention may be selected from the low melting forms of the encapsulating materials previously mentioned above. A particularly useful class of thermoplastic materials for use in such embodiments are the waxes and especially beeswax (solidification point 60.5 to 62°C). Such waxes, in addition to providing a means of accomplishing the objectives set forth above may also, in some instances, be a useful ingredient in the composition or provide it with desirable characteristics such as body.

Thermoplastic capsules for use in the above embodiments may be readily prepared by known processes. In one such process, the thermoplastic encapsulating material is dispersed in an external phase which is a nonsolvent for both the encapsulating material and the material to be encapsulated. The system is heated to melt the encapsulating material and the material to be encapsulated is then added. As the system is cooled, the encapsulating material solidifies around the material to be encapsulated and thereby forms the capsules. The capsules, thus produced, are removed from the system by filtration.

As can be appreciated from the above, the concepts of the present invention are applicable to a broad range of compositions. Such compositions may be ilustrated by the following shaving cream formulation which is prepared as set forth below.

An 8.2% aqueous hydrogen peroxide solution (the oxidant) is encapsulated in beeswax by methods such as set forth above, to provide capsules having an average diameter of at least 2000 microns. The capsules are uniformly dispersed in a shaving cream formulation having the following composition by weight:

	Parts
1 - phenyl - 5 - ethyl - 2 - thiobarbituric acid (the reductant)	5.00
Ammonium molybdate tetrahydrate (the catalyst)	0.06
98% triethanolamine	11.20
Triple pressed stearic acid	7.55
Stripped coconut fatty acids (see below)	0.96
Potassium hydroxide pellets (86%)	1.07
Sodium lauryl sarcosinate	3.22
Stearamide	0.96
Perfume	0.39
Distilled water	66.09

(i.e. those fatty acids derived from coconut oil which have been processed so as to remove acids which are lower in molecular weight than lauric acid).

The capsules are added to the formulation in amounts such that there will be about 1 part by weight of the aqueous peroxide solution present for each 3 parts of the shaving cream formulation.

In using the above composition for shaving, an amount of it, sufficient for one shave, is applied to the hand and rubbed to rupture the capsules and release the aqueous peroxide solution. Upon release, the peroxide reacts with the 1 - phenyl - 5 - ethyl - 2 - thiobarbituric acid reductant to elevate the temperature of the composition. The rubbing is continued until the melted capsule fragments are dispersed in the composition and the resulting hot shaving cream is then applied to the beard.

If it is desired to make the above shaving formulation self-foaming as well as self-heating, this can be readily accomplished by adding a foam-generating material, such as described above, to the composition. Usually, the use of about 0.75 to about 2.0 gram molecular weights of the foam-generating material per 1000 gms of the aqueous composition will provide a useful foam. If it is desired to sustain the heat-generating action, this is accomplished by using an excess of the heat-generating components and initially rupturing that portion of the capsules which will bring the composition or a portion of it to a temperature which will gradually melt the remaining unruptured capsule.

In packaging the compositions of the present invention, it is generally not advisable to use squeeze type tubes because a certain portion of the capsules in the unused portion of the composition will be ruptured during the dispensing operation. If it is desired to package the compositions in bulk, it is best to use wide-necked jars which will enable the user to gingerly remove the portion he wishes to use without rupturing the capsules in the unused portion of the composition.

When using such bulk packaging, the viscosity of the compositions should preferably be such that the capsules will remain uniformly suspended in the composition until time of use and will not require stirring. Such suspension of the capsule may be achieved by employing in the compositions one or more

of the many thickening and suspending reagents which are commercially available.

The compositions of the present invention are particularly useful in single use packets. Preferred forms of single use packets are shown, by way of example, in the accompanying drawing in which Figure 1 is a perspective front view of a first packet and Figure 2 is a similar view of a second packet.

Referring to Figure 1, the packet 1 comprises a completely enclosed chamber 3 which is bounded by resilient side walls 4 which are sealed to each other along their peripheries. The chamber opens into a narrow dispensing channel 7 which is closed off by a tear-off cap 9. Within the chamber, there is provided a shaving cream composition 5 containing a plurality of first 11 and second 13 capsules. The first capsules 11 contain a heat-generating material which is reactive with another heat-generating material which is disposed in the shaving cream composition and the second capsules 13 contain a foam-generating material. In use, the side walls 4 of the packet 1 are pressed together to rupture the capsules 11 and 13. When the capsules 11 and 13 are ruptured and the composition becomes hot and begins to foam, the tear-off cap is removed. Under the pressure of the foam-generating material, the hot shaving composition is forced out of the packet 1 for use.

In Figure 2, there is shown a packet similar to that shown in claim 1, except that it is encased by a rigid protective sheath 15 which prevents the accidental rupturing of the capsules 11 and 13. The sheath 15 is provided with an opening 17 in its top through which the capped dispensing channel 7 protrudes and in the side-walls of sheath 15 there are positioned openings 21 which provide access to the resilient side walls 4 so that they may be pressed together during use to rupture the capsules 11 and 13.

It should be appreciated that in single use packets, such as shown in Figures 1 and 2, the foam-generating materials, when desired, may be disposed directly in the composition. Similarly, the heat-generating materials and the foam-generating materials may be disposed in single large frangible capsules which are positioned contiguous to the composition.

In embodiments of the present invention where the heat-generating component and the foam-generating material are inert with respect to each other, it should be understood that they may be encapsulated in the same capsules.

WHAT WE CLAIM IS:—

1. A self-heating pharmaceutical or cosmetic composition, comprising a vehicle and a first and a second heat-generating component maintained separately from one another but which when in contact with one another effect an exothermic reaction; at least one of said components being encapsulated into a plurality of frangible capsules which are dispersed in said vehicle, the encapsulating material for said capsules being both inert and impermeable with respect to the vehicle and the heat-generating components.

2. A self-heating composition according to claim 1, wherein the encapsulating material has a melting point which is at or below the temperature which is generated in the composition when said heat-generating components react.

3. A self-heating composition according to claim 1 or 2, which includes a foam-generating material, which is insoluble or only slightly soluble in the vehicle, said foam-generating material being a liquid at room temperature and having a boiling point which is below the temperature which is generated in the composition when said heat-generating components react.

4. A self-heating composition according to claim 3, wherein said foam-generating material is encapsulated into a plurality of frangible capsules which are dispersed in said vehicle, the encapsulating material for said capsules being both inert and impermeable with respect to said vehicle and said foam-generating material.

5. A self-heating composition according to any of the preceding claims, which is capable of evolving heat over a prolonged period, wherein said heat-generating components are present in amounts in excess of that which is required to bring the composition to a desired specified temperature and the encapsulating material for said capsules has a melting point at or below said specified temperature, whereby upon rupturing at least that portion of the capsules which will bring at least a portion of the composition above said melting point, the unruptured capsules will gradually melt over a prolonged period and continuously release additional quantities of the heat-generating components.

6. A self-heating composition according to claim 5, which includes a foam-generating material which is encapsulated into a plurality of frangible capsules which are dispersed in said vehicle, the encapsulating material for said capsules being both inert and impermeable with respect to said vehicle and having a melting point at or below said specified temperature, whereby said capsules will be melted over a prolonged period and continuously release additional quantities of said foam-generating material.

7. A self-heating composition according to any one of claims 1 to 4, which rises gradually to a specified temperature over a prolonged period, wherein said heat-generating components are encapsulated in a plurality of encapsulating materials which have sequentially higher melting points which are above the temperature at which the composition is

normally stored and at or below said specified temperature.

8. A self-heating composition according to claim 7, which further evolves heat over a prolonged period when said composition reaches the specified temperature, wherein the composition comprises said heat-generating components in amounts in excess of that required to elevate the composition to said specified temperature, the excess of heat-generating components being encapsulated in the encapsulating material which has the highest melting point in said plurality of encapsulating materials.

9. A self-heating composition according to any of the preceding claims, which is a shaving cream.

10. A self-heating composition according to claim 1, substantially as hereinbefore described.

11. A single-use packet containing a self-heating composition, said packet comprising an enclosed chamber in which a composition as defined in any of the preceding claims is dispersed, said chamber being bound by at least one resilient wall and having a dispensing opening which is closed off by a removable closure.

12. A single-use packet containing a self-heating composition, substantially as hereinbefore described with reference to Figures 1 and 2 of the accompanying drawing.

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1308565 COMPLETE SPECIFICATION

1 SHEET

This drawing is a reproduction of the Original on a reduced scale

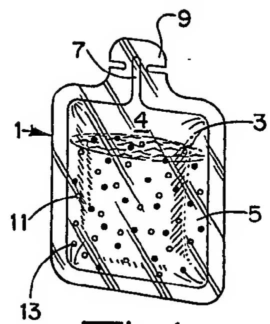

Fig. 1.

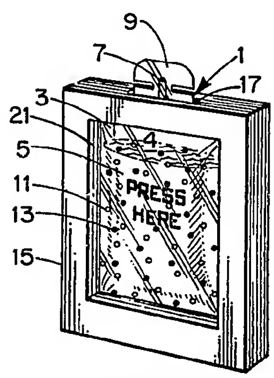

Fig. 2.